

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



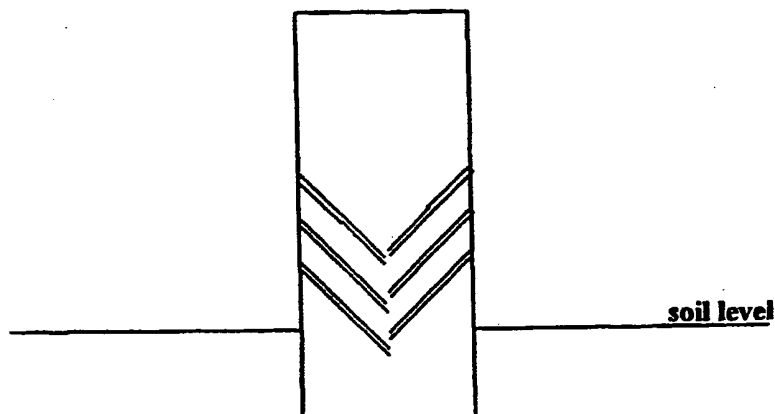
(43) International Publication Date
28 June 2001 (28.06.2001)

PCT

(10) International Publication Number
WO 01/46085 A2

- (51) International Patent Classification⁷: C04B 41/00 (81) Designated States (*national*): AU, BA, BG, BR, CA, CN, CU, HU, IN, JP, MK, NO, PL, RU, US, YU.
- (21) International Application Number: PCT/RO00/00025 (84) Designated States (*regional*): Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR).
- (22) International Filing Date:
20 December 2000 (20.12.2000)
- (25) Filing Language: English Published:
— Without international search report and to be republished upon receipt of that report.
- (26) Publication Language: English
- (30) Priority Data:
99-01376 23 December 1999 (23.12.1999) RO
00-01019 16 November 2000 (16.11.2000) RO
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.
- (71) Applicant and
(72) Inventor: COȘOFREȚ, Sorin, Cezar [RO/RO]; Str. Sfântul Atanasie nr.30, R-Iași, județ Iași, 6600 (RO).

(54) Title: COMPOSITION FUNCTIONING AS A BINDING AGENT AND AS A COMBAT FACTOR AGAINST ASCENSIONAL HUMIDITY IN BUILDING AND METHOD TO APPLY



(57) Abstract: The invention consists of colloidal emulsion composition used for the protection of building elements against the damaging action of water penetrating them by capillarity or hydrostatic pressure. The emulsion may be also utilized as a binding agent for the building element crumbling by the action of meteorological, chemical and biological factors. The emulsion will be used by injection, in order to achieve a horizontal hydroinsulation, through orifices made either in one side or both sides of the building element, the distance among orifices being correlated with the emulsion diffusion radius around the orifice, so that no area among orifices remain unimpregnated with the emulsion.

WO 01/46085 A2

**COMPOSITION FUNCTIONING AS A BINDING AGENT
AND AS A COMBAT FACTOR AGAINST ASCENSIONAL HUMIDITY
IN BUILDING AND METHOD TO APPLY**

Description

The invention consists of a colloidal composition used for the protection of building elements against the damaging action of water penetrating them by capillarity of hydrostatic pressure.

As typical, but not limitative examples, the composition may be used in the following situations:

- ♦ soil water penetration in old buildings (main causes: either deterioration or even absence of insulation);
- ♦ the presence of water natural sources in the proximity of the building foundations;
- ♦ the deterioration of the exterior insulation of underground building elements;
- ♦ the presence of other water sources (water pipes, water tower, etc) producing by different causes water penetration into the building by hydrostatic pressure and capillarity;
- ♦ surface covering for structure elements in order to protect them.

The composition may be also used for restoration of old buildings as a binding agent of building materials crumbled by the action of meteorological, chemical and biological factors.

There are several procedures to protect the structure elements against the soil water penetration, generally based on electro-osmosis in different practical variants (with or without a source of auxiliary electric power). So it is possible to utilize a system consisting of an electroconductive dye, applied to the whole surface of the wall and a series of cylindrical anodes inserted into the bottom of the same wall, together with a source of electric power (Patent Ro 64969). Patent Ro 91966 also presents a variant of electro-osmosis functioning without any auxiliary electric power.

The main disadvantage of the electro-osmosis procedures (either active or passive) is that their effect is positive as long as the devices implanted in the masonry

are kept therein. The dampness in the wall appears again with the deterioration or removal of these devices.

The patent Ro 75304 presents an organic compound for building elements protection against the water soakage on the basis of diisocyanates.

The compound has the disadvantage of necessitating the use of organic solvents (methylisobutyl ketone, toluen, etc.) which increase its cost, inflammability and toxicity.

The hydrophobic properties of silanes, siloxanes and silicones are also known. So, in the Patent RO 107266 is present a compound of anticondense varnishes based on epoxi resins and methylphenylsilicone, also utilizable to combat ascensional humidity by electro-osmosis.

The tehcnical problem this invention solves is that the building element affected by dampness or destined for restoration is impregnated by injecting a colloidal emulsion in the wall; after impregnating, the colloidal emulsion components become a jelly obstructing the pores of the building element and at the same time playing the role of a binder of the building material. In time, the building element dries out, and the jellied zone forms a hydrophobic, impermeable layer, which stops the water capillarity transport to the building upper parts.

In accordance with the invention, the emulsion for injection in building elements consists of:

(a) an active component, which bloks up the same time consolidates the building element, in a concentration of 5-60% consisting from:

a₁- colloidal silica;

a₂- alcali-silicates (lithium silicate, sodium silicate, potassium silicate) or mixture of them;

a₃-an ammonium quaternary silicate or mixtures of similar silicates;

a₄-an alkylalcoxisilane or mixtures of alkylalcoxilanes;

a₅-a siloxane or mixtures of siloxanes;

a₆-a silicone or mixtures of silicones;

a₇-an alkyl orthosilicate or mixtures of similar orthosilicates;

a₈-silanoles, siloxanes siloxanoles salts;

a₉- all mixtures, in any proportion, composed of at least two of aforementioned components(a₁,a₂,a₃,a₄,a₅,a₆,a₇, a₈);

(b) a viscosity regulator in a concentration of 0-5%;

(c) a nonionic, anionic, cationic or ampholyte emulsifier in a concentration of 0-15%;

(d) a pH regulator, preferably a buffery system or an alkali in a concentration 0 -5 %;

(e) other dispersed systems of polymers and/or oligomers in a concentration of maxim 10%;

(f) other components in a maximum concentration of 2%;

(g) a solvent or a system of solvents, so that $a+b+c+d+e+g=100$.

The active component:

a_1 and a_2 -a compound in the form of aqueous solution or dispersion of colloidal silica or an alcali silicates with the formula (a_{31}): $M_2O \times n SiO_2$, where M is Li, Na or K and n a natural number or a mixture of such silicates or wherein, and optionally, many be also present in solution Mg, Ca, Ba, Sr, Zn, Al, Fe, ions, which in the presence of a complexants operate as jellification agents;

a_3 -ammonium organic quaternary silicates with the general formula (a_{31}): $((R_1R_2R_3R_4)N)_2O \times nSiO_2$, which are usually obtained by the reaction of an ammonium quaternary salt which silica sol or by the reaction of silica sol with an amine and with ethylene oxide.

In the formula a_{31} : R_1, R_2, R_3, R_4 may be identic radicals or different ones to no more than 25 carbon atoms, originating in an alkane, cycloalkane, alkene, arene with or without lateral chain, alcohol, halogen derivative, alcohol, amine, ester.

As examples of such ammonium quaternary silicates are the following ones: tetramethylammonium silicate, methyltriethanolammonium silicate, tetraethanol-ammonium silicate etc.

a_4 -The utilized silanes may be: monoalcoxysilanes, dialcoxysilanes or trialcoxysilanes, or their mixtures.

The utilized *trialcoxysilanes* correspond to the formula a_{41} : $RSi(OR')_3$ where R is a monovalent organic radical which has up to 25 carbon atoms and results from an alkane, cycloalkane, alkene, arene with or without lateral chain, halogen derivative, alcohol, amine, ester and R' is a rest alkyl having to five carbon atoms. As examples of R hydrocarbon radicals we mention: 3,3,3 trifluoropropyl, o-,m-,p-chlorophenyl, 3aminopropyl, N-cyclohexyl-3aminopropyl, methacryloxypropyl, acryloil, methacryloil, methacryloxyethyl, etc.

As examples of radicals represented by R, there are the atoms of hydrogen, methyl, ethyl, n-propyl, isobutyl, tertbutyl, etc.

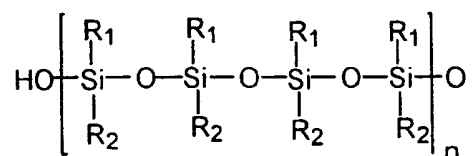
Mixtures of two or more trialkoxysilanes may be also utilized in the realization of the composition.

Dialcoxysilanes correspond to formula (a₄₂): $RR''Si(OR')_2$, where R' has the same significance as before, and R and R'' are identic or different monovalent organic radicals. R has the afore mentioned significance, and R is also a monovalent radical, which meets the same conditions as R. Here are some examples of hydrolyzable dialcoxysilanes: dimethyldimethoxysilane, dimethyldiethoxysilane, dipropyl dimethoxysilanes, dipropyldiethoxysilanes, methylphenyldimethoxysilane, N-aminoethyl-aminopropylmethyldimethoxysilane, N-cyclohexyl-aminopropylmethyl dimethoxysilane.

Dimethyldimethoxysilane is preferable. Mixtures of two or more dialcoxysilanes may be also utilized.

Monocoxysilanes correspond to formula $RR''R'''SiOR'$, R, R'', R', have the above-mentioned significances, and R''' is also a monovalent radical that meets the same conditions as R and R'' being either identic with or different from them.

a₅-The utilized siloxanes have the formula a₅₁:



where R₁ and R₂ represent identic or different radicals meeting the same conditions as R in formula a₄₁. It is preferable that R₂ have no more than 5 carbon atoms, and the ethyl radical also preferably.

a_n-The utilizable silicones correspond to formula: (a₆₁): $RSiO_a(OR')_b$, where R and R' represent identical or different organic radicals (coming from an alkane, cycloalkane, arene (with or without lateral chain) to maximum 25 carbon atoms which may also have substitutes (halogen, hydroxyl, amino, ester); a, b real numbers between 0 and 3, preferably a between 0.75 and 1.5, and b between 0.01 and 0.07, and the molecular mass of the silicone being more than 4000, and containing free groups of hydroxyl at the ends of silicone chain.

Also R' may be represented by hydrogen.

In order to prepare the emulsion it is possible to utilize one silicone or a mixture of silicones with a molecular mass in the limits of 4000 and 400000.

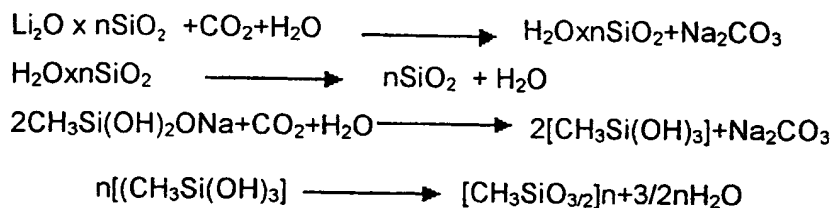
a_7 - alkyl orthosilicates are obtained by the reaction of silicon tetrachloride with the respective alcohols. The alkyl silicates correspond to the formula: $\text{Si}(\text{OR})_4$ where R is a monovalent radical, identical or different, resulted from an aliphatic hydrocarbon (alkane, cycloalkane) to nine carbon atoms. As examples of such silicates there are: methyl silicates, n-propyl silicate, ethyl silicate, n-butylsilicate.

a_8 - the salts of silanols, siloxanes, siloxanols are obtained by substitution of a hydrogen atom from the OH group combined with the silicon atom, for an alkali metal. It is possible to use salts resulted from alcoxysilanes and siloxanes aforementioned. Preferably, it will be utilized sodium methylsilicone with formula:

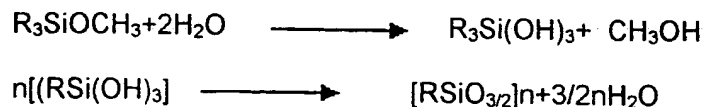


a_9 - any combination, in any proportion, formed by at least two components aforementioned ($a_1, a_2, a_3, a_4, a_5, a_6, a_7, a_8$).

By injecting into wall, silicate-based (inorganic or organic) variants, or their derivatives, generate silicic acid or their substituted derivatives, which, by condensation with water educing, form macromolecular structures blocking up the masonry pores according to the following expression:



The variants based on alcoxysilanes hydrolyze forming silanols and silandioles, unstable water educing substances, which form siloxanes and/or silicones according to the undermentioned reactions:



The siloxane-based variant undergoes a reaction of a linear condensation or of a cyclic one with water elimination and silicones formation.

In the silicon-based variant, the hydrophobic effect is firstly obtained by elimination of water from the composition to be injected, as is well known the

hydrophobic property of silicones, and secondly, by condensation processes among polymer chains.

(b) -as a component (b), it is possible to utilize a substance or a mixture of such substances, stable in ambient base, viscosity diminishing. This is very important to let the emulsion penetrate through the pores of the building element. Were a composition of high viscosity utilized, just at an injection under pressure of the composition, this would not uniformly impregnate the whole building element, leading to a defective treatment with the possibility of reappearance of the ascensional humidity after the treatment.

As (b) component are utilizable:

- ♦ cellulose derivatives: carboxymethylcellulose, methylcellulose, hydroxyethyl cellulose, etc;
- ♦ polyvinyl alcohol;
- ♦ polyacrylates and polymethacrylates alkali salts like: sodium polyacrylate, sodium polymethacrylate, etc;
- ♦ sodium and ammonium salts of carboxylate polymers;
- ♦ silanol and siloxanol salts like sodium methylsilicone.

It is preferable to utilize compositions using salts of silanols and siloxanols functioning also as an active substance.

(c) In order to obtain the composition, it may be utilized a nonionic, anionic, cationic or an ampholyte emulsifier, for maintaining the stability of dispersion. To achieve and use emulsifiers is necessary only in a_1 , a_2 , a_3 cases or in variants which have these possibilities, for the other variants an emulsifier is not absolutely necessary.

Anionic emulsifiers to be used:

- ♦ alkaline salts (Na, K) of alkylbenzenesulfonic acid;
- ♦ alkyl-sulfates with hydrocarbon rest of 5-20 C atoms and alkylethers-sulfates containing to 40 units of ethylene oxide or propylene oxide;
- ♦ alkaline salts (Na, K) of casein;
- ♦ algin acid and alginates of alkaline metals or alkaline earths;
- ♦ polyphosphates of alkaline metals or alkaline earths;
- ♦ sulfatate alcohols, type $R-OSO_3Na$, where R is an alkyl, aryl, or alkylaryl radical with 5 to 25 carbon atoms;

- ♦ sulfatate esters, type $RCH_2CH(SO_3Na)COOR$, where R is a radical having to 15 carbon atoms;
- ♦ esters or sulfosuccinic acid with monoalcohols or alkylphenols, where, optionally, monoalcohols or alkylphenols may be ethoxylated with 1-40 units of ethylen oxide or propylen oxide;
- ♦ alkaline or ammonium salts of alkyl, alkylaryl, arylalkyl phosphates with 6 to 20 C atoms in the hydrocarbon rest;
- ♦ alkyl, alkylaryl, arylalkyl phosphates (or their alkaline salts) with 6 to 20 C atoms in the hydrocarbon rest and containing to 40 units ethylene oxide or propylene oxide per molecule;
- ♦ ether alcohols and sulfonate ether phenols (for example: $RO(CH_2CH_2O)_xCH_2CH_2OSO_3Na$), where R is an alkyl, aryl, or alkylaryl radical with 5-25 C atoms;
- ♦ aliphatic sulfonic acids (RSO_3Na) with a hydrocarbon chain, 8 to 25 C atoms long, where R may still contain stable functional groups at hydrolysis in aqueous medium;
- ♦ sulfonic acids of dialkylated naphthalene ($R_2C_{10}H_5SO_3Na$);
- ♦ alkylaryl sulfonates with formula ($RC_6H_4SO_3Na$), where R is saturated or unsaturated hydrocarbon rest of maximum 25 C atoms;
- ♦ sulfonic esters of dicarboxylic acids (sulfonic esters of succinic acid);
- ♦ anionic emulsifiers, type alcoylate, described in W09907673 (EP.1005452);
- ♦ esters of higher acids with hydroxysulfonic acids ($RCOOCH_2CH_2SO_3Na$);
- ♦ acyl derivatives (amides) of superior acids with aminosulfonic acids ($RCON(CH_3)CH_2CH_2SO_3Na$);
- ♦ phosphatides;
- ♦ alkaline earths or ammonium salts of carboxylic acids with 6-20 C atoms, where the hydrocarbon rest may be alkyl, aryl, or arylalkyl.

Nonionic emulsifiers to be used:

- ♦ alcohol ethers – obtained as condensation products of ethylene oxide with higher alcohols, where alcohol hydrocarbon rest may have linear or ramified catena and 7-25 C atoms and the number of ethylene oxide units is under 50;

ex: $C_{18}H_{37}O(CH_2CH_2O)_xCH_2CH_2OH$;

- ♦ phenolethers – obtained as condensation products of ethylene oxide with substituted phenols in nucleus with an aliphatic catena with 5-20 C atoms and ethylene oxide units number less than 50: ex: $RC_6H_4O(CH_2CH_2O)_xCH_2CH_2OH$;
- ♦ A-B-A compounds, where each A represents an oligo rest or a polyester of a hydrocarboxylic acid, and B is a chain of polyoxoalkylene type;
- ♦ emulsifiers containing sulfoxy groups described in patent GB 2077620 (ex. tertiary-dodecyl-sulfoxy-polyethylene glycols):
 - ♦ polyoxyethylenesters of fatty acids;
 - ♦ polyoxyethylenalkylethers and polyoxyethylenalkylphenylethers;
 - ♦ partially esterified polyglycerol;
 - ♦ natural substances like: lecithin, lanolin, saponin, cellulose alkylated derivatives with at most 4 C atoms in alkyl rest;
- ♦ polyoxyethylenglycols modified in conformity with GB-P No. 2,093,783, DE-PS No. 3,109,317, U.S. Pat. No. 4,258,169, U.S. Pat. No. 4,257,995, DE-PS No. 2,932,175, DE-OS No. 3,111,562, either modified with esters of phosphoric acid (DE-PS No. 19,456) or modified with sulfonic acids (EP 19486);
- ♦ ethoxy products of monoalcohols, monoamines, monomercaptans, alkylmonophenols, in conformity with DE-OS No. 1,081,225;
- ♦ cyclic emulsifiers in conformity with DE-OS No. 3,062,328 / DE-OS No. 3,060,545;
 - ♦ amines and amides derived from fatty acids;
 - ♦ esters of fatty acids with alcohols, ethylenglycol, polyethylenglycol, glycerine, polyglycerine, propylenglycol, sorbitol, pentaerythritol, saccharose;
 - ♦ polyethylenglycol condensation products (or polyethylenglycol oligomers) with alcohols, thioalcohols, fatty acids, amines and amides of fatty acids, polypropylenglycol, ethylenediamine, alkylphenols, condensed phenols, abietic acid, condensed amines and polysiloxane;
 - ♦ propylenglycol condensation products with alcohols, fatty acids, and their derivatives (amines, amides, esters);
 - ♦ higher alcohols with 8-20 C atoms, obtained by "oxo" synthesis;
 - ♦ hydroxyethylcellulose, methylcellulose, gelatine, polyoxyethylenpolypropylen glycolether, salts of aminated lignin;
 - ♦ polyvinylalcohol with a polymerization degree to 5000.

- ♦ glycolic ethers with hydrocarbon radicals having 8-20 C atoms, resulting from an alkyl rest or aryl one:
 - ♦ colofony esters with glycerol;
 - ♦ copolymers of ethylene oxide or propylene oxide;
 - ♦ alkylpolyglycolethers having to 50 units resulted from ethylene oxide and alkyl rest to 20 C atoms:
 - ♦ alkylarylpolyglycolethers with at most 50 units resulted from ethylene oxide and with an alkyl rest or aryl rest to 20 C atoms;
 - ♦ ethylene oxide or propylene oxide oligomers with 200 units maximum;
 - ♦ addition products of ethylene oxide or propylene oxide at amines with alkyl radical at most 20 C atoms:
 - ♦ carboxylic acids with 6-24 C atoms per molecule;
 - ♦ alkylpolyglycosides with formula R-O-Zi, where R is a saturated, aliphatic or aromatic, radical with 6-20 C atoms, and Zi is an oligosaccharide, with at most 10 units of monosaccharide;
 - ♦ esters of sugars with alphahydroxycarboxylic acids and lactides in conformity with DE 19829305;
 - ♦ polyoxyethylenglycols and monoalkylethers of polyoxyethylenglycol;
 - ♦ cyclic emulsifiers in conformity with DE-OS 3062328, 3060545;
 - ♦ polyoxypropylenglycol reaction products with ethylene oxide;
 - ♦ polyoxyethylenpolyoxypropylenglycol soluble in water;
 - ♦ natural substances or derivatives like: lecithin, lanolin, saponin, cellulose, carboxyalkylcellulose with alkyl rest of 5 C atoms at most, etc;
 - ♦ modified organopolysiloxanes containing at most 40 units ethylene oxide per molecule:
 - ♦ organopolysiloxanes modified, in conformity with US 6060619, by the reaction of carboxyl group from the siloxane molecule with the hydroxyl group of the lactic acid.

Cationic emulsifiers for use:

- ♦ salts of primary, secondary, tertiary, amines containing 8-24 C atoms radicals with acetic acid, sulfuric acid, hydrochloric acid, phosphoric acid;
- ♦ alkylamines with 6-20 C atoms;

- ♦ ammonium quaternary salts of alkylarylamines or arylalkylamines with at most 24 C atoms per molecule, with acetic acid, sulfuric acid, hydrochloric acid, phosphoric acid.;
- ♦ salts of alkylpyridine, alkylimidazole, alkylloxazole with alkyl rest of at most 20 C atoms, with acetic acid, sulphuric acid;
- ♦ salts of alkylpolyamines.

Ampholytic emulsifiers to be used:

- ♦ amino acids substituted, or not substituted, having at most 25 C atoms per molecule;
- ♦ betaine, alkylbetaine and alkylimidazole betaine with alkyl radical with at most 18 C atoms.

(d) a pH regulator, preferably a buffery system or abase, in order to maintain the emulsion pH at a certain value, generally between 7 and 11,5. Such a value of pH is necessary because the composition is stable in a base ambience.

Therefore it is possible to use organic or inorganic bases, provided that they do not affect the emulsion stability. Buffer mixtures or base substances will be particularly used like: alkaline carbonates or bicarbonates (sodium or potassium), sodium phosphate, ammonium acetate, alkaline polyphosphates, sodium molybdate, etc. Among the buffery organic substances, amines are useful as for instance: diethylamine, ethylendiamine, monoethanolamine, triethanolamine, morpholine, 2-amino-1-propanol, etc.

(e) Other dispersal systems of additional polymers and oligomers play a part in ensuring a good elasticity and/or plasticity of the coating resulted from the solidifying of the emulsion injected into the building element. So, in the emulsion composition may be added resins: epoxy, phenol, polyester, vinylester, polyurethane, polyamide, furan, acrylic, polyethylene, polypropylene, polystyrene, polyvinylacetate resins, etc.

The part of the dispersed systems of polymers and oligomers is to realize a proper plasticity, even the elasticity of the constituting layer. In the case of building elements unexposed at permanent or repetitive vibrations it is preferable to inject an emulsifiable resinless composition forms a hard, but breakable layer.

In the case of building elements exposed to permanent vibrations, it is preferred a composition with added emulsifying resins, which form a more plastic layer, even an elastic one.

(f) Other components in a maximum 2% concentration include fungicide, bactericide, colouring, catalytic, aromatic corrosion inhibiting substances, etc.

(g) A solvent or a system of solvents, so that $a+b+c+d+e+f+g=100$. Demineralized water is preferred as a solvent. At compositions containing alkyl orthosilicates it is to be used as a solvent a volatile alcohol because they are not stable in water.

The composition is realized by emulsifier dispersion (if used) into the active component, by stirring, then a small quantity of solvent is added, also by stirring, to obtain a viscous concentrate substance. Components b, d, e, f, are added by stirring and afterwards is the concentrated stuff diluted by stirring until the desired percentage of dry substance is obtained.

In order to inject the composition in the treated wall, orifices are made with a diameter of 12-20 mm, at a distance of 10-20 cm among them, either horizontally or at any angle to the vertical surface of the wall, in both sides of the wall or on one side. When the orifices are made in both sides of the wall (in case of walls above the soil level), they must penetrate to the mid-breadth of it (fig 1 and fig 2); when they are made on one side (in case of a walls under the soil level or in case of walls above the soil level but with difficult acces to one side of them), then the orifices must be 3-4 cm deep to the other side (fig. 3 and fig. 4). Afterwards, from a reserve, by means of a distribution system, is the emulsion injected in the wall. It is also possible to utilise devices of injecting under pression the composition.

In the first phase the wall will absorb a larger quantity of emulsion, but with the beginning of the silica jelly formation the quantity of the emulsion absorbed by the wall diminishes, and by the masonry pores blocking and by the waterproof layer formation the emulsion is not any more absorbed. At this moment the treatment against dampness is ended. The orifices are stopped with a mixtures of cement, sand and emulsion; then if necessary, teh outside is remade.

The invented composition functioning as a binding agent and as a combat factor against ascensional humidity has the following advantages:

- ◆ it ensures water proofing the building elements by blocking the masonry pores in the treated area;
- ◆ it strengthens the masonry by its binding effect on old brakeable materials;

- ♦ it definitively remedies, and dampness doesn't appear any more at higher levels;
- ♦ it completely eliminates humidity because of the capillarity;
- ♦ it improves the termic transfer resistance of the treated masonry;
- ♦ it offers an efficient resistance to microorganisms aggression;
- ♦ the emulsion composition to impregnate is compatible with usual construction materials:

- ♦ the product is ecological.

Examples of composition preparation:

Example No. 1

The emulsion with 13% Lithium silicate concentration prepared as follows: to 100 m.p. (m.p.=mass parts) Lithium silicate (concentration 30%) and $\text{Li}_2\text{O}:\text{SiO}_2$ ratio of 1:2.2 by stirring, it is added: 10 m.p. methyl cellulose as emulsifier and reduction factor of viscosity, 5 p.m. monoethanolamine, 25 m.p. acrylic polymers emulsified in water, 0.001 m.p. alizarin yellow. Continuously, by stirring, it is added 95 m.p. demineralized water. After water addition, stirring is continued at 4000 r.p.m. for half an hour. The resulted product is filtered by kieselguhr.

Example No. 2

The emulsion with $\approx 18\%$ colloidal silica concentration prepared as follows: to 100 m.p. (m.p.=mass parts) silica sol (concentration 35%) by stirring, it is added: 10 m.p. methyl cellulose as emulsifier and reduction factor of viscosity, 5 p.m. monoethanolamine, 25 m.p. acrylic polymers emulsified in water, 0.001 m.p. alizarin yellow. Continuously, by stirring, it is added 95 m.p. demineralized water. After water addition, stirring is continued at 4000 r.p.m. for half an hour. The resulted product is filtered by kieselguhr.

Example No. 3

The emulsion is like that in example 1, but it starts with 100 m. p. lithium and potassium silicates mixture obtained by mixing 40 m.p. Li silicate, 30% concentration, and $\text{Li}_2\text{O}:\text{SiO}_2$ ratio of 1:2.2 and 60 m.p. potassium silicate ($\approx 41\%$) at 1.41g/cm^3 density and a $\text{K}_2\text{O}:\text{SiO}_2$ ratio of 1:2. Sodium palmitate is used as emulsifier. The resulted emulsion has approximately 15% soluble silicates.

Example No. 4

The emulsion is obtained like in example 1, but it starts with 100 p.m. mixture of Li silicate and colloidal silica obtained by mixing 40 p.m. Li silicate, concentration 30% and $\text{Li}_2\text{O}:\text{SiO}_2$ ratio of 1:2.2 and 60 p.m. colloidal silica at 30% concentration and as emulsifier is used ammonium stearate.

Example No. 5

Emulsion as in example 1, but it starts with 100 p.m. mixture of Li silicate and sodium methylsilicate, obtained by mixing 40 p.m. Li silicate, 30% concentration and $\text{Li}_2\text{O}:\text{SiO}_2$ ratio of 1:2.2 and 60 p.m. sodium methylsilicate, 30% concentration and polyvinyl alcohol as emulsifier.

Example No. 6

To 100 parts of tetraethanolammonium silicate are added by stirring 10 p sodium methylsilicate, 5 p.m. polyethylenglycol-based emulsified (Arlypon IT 16), 10 p hydroxyethylcellulose, 20 p ethene-acrylic acid copolymers dispersion, 5 p morpholine, 0.001 p colouring matter, 300 p demineralized water is added. After 1/2 hour long stirring, the product is filtered.

Example No. 7

100 p tetraethanolammonium is mixed in 100 p potassium silicate solution (41%). It is continued as at example 5 and finally 450 ml water are added.

Example No. 8

By stirring are mixed 10 m.p. emulsifier consisting of ester of oleic acid with isethionic alcohol and 250 m. p. buthyltriethoxysilane, 5 m.p. ethylendiamine, 20 m.p. acrylic emulsion, 0.001 m.p. basic azoic colouring matter and 350 m.p. disionized water.

The stirring is continued at increased velocity 1/2 hour long in order to obtain a fitted dispersion.

Examples 9-12

To act as in example 7 with the only difference that instead of buthyltriethoxysilane is used one of the following variants:

9) dimethyldimethoxysilane

10) methacryloxytrimethoxysilane

11) dipropyldimethoxysilane mixed with N-cyclohexyl-aminopropylmethyldimethoxy silane.

12) tripropylmethoxysilane mixed with N-cyclohexyl-aminopropylmethyldimethoxy silane.

Example 13

Two parts sodium laurate- emulsifier and 70 parts water are mixed, by vigorously stirring, with 100 parts dimethylpolysiloxane and 30 units siloxane. By addition of NaHCO_3 , 30 parts polyvinyl acetate emulsion, 0,001 parts alizarin yellow. pH is set at 7,5. Continuously stirring, 150 m.p. demineralized water is added to. After water addition, stirring is continued at 4000 r.p.m. for half an hour. The resulted product is filtered by kieselguhr.

Example 14

To 100 m.p. ethyl orthosilicate are added by stirring 10 m.p. hydroxyethylcellulose, 5 m.p. morpholine, 0,001 colouring matter. The mixture is diluted by 300 ml ethyl alcohol. Stirring is continued for half an hour. The product rezulted is filtered by kieselguhr filter.

Example 15

To 100 m.p. sodium methylsilicate are added by stirring 10 m.p. hydroxyethylcellulose, 10 m.p. morpholine, 0,001 m.p.colouring matter. The mixture is diluted by 300 ml demineralized water. Stirring is continued for half an hour. The product rezulted is filtered by kieselguhr filter.

Example 16

To 100 m.p. polydimethylsiloxane with approx. 200000 molecular mass (having hydroxyl groups at the ends of the silicone chains) are added, by stirring, 15 p.m. emulsifier Nekali (sodium salt of sulfonic acid of alkylate naphthalin). By stirring are added: 10 m.p. polyvinyllic, 5 m.p. triethanolamine, 20 m.p. acrylic emulsion, 0,001 m.p. basic azoic colouring matter and 350 p.m. disionized water. Stirring is continued at increased velocity 1/2 hour long in order to obtain a fit dispersion.

**COMPOSITION FUNCTIONING AS A BINDING AGENT
AND AS A COMBAT FACTOR AGAINST ASCENSIONAL HUMIDITY
IN BUILDING AND METHOD TO APPLAY**

Claims:

1. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them; it is characterized by the fact that, in order to realize the consolidation of the building elements and the removal of the ascensional humidity, it is used a colloidal emulsion containing:

(a) an active component, which blocks up the masonry pores and concomitantly consolidates the building element, in a concentration of 5-60%, chosen from:

a₁- colloidal silica;

a₂- alkali-silicates (lithium silicate, sodium silicate, potassium silicate) or mixture of them;

a₃-an ammonium quaternary silicate or mixtures of similar silicates;

a₄-an alkylalcoxysilane or mixtures of alkylalcoxilanes;

a₅-a siloxane or mixtures of siloxanes;

a₆-a silicone or mixtures of silicones;

a₇-an alkyl orthosilicate or mixtures of similar orthosilicates;

a₈-silanoles, siloxanes siloxanoles salts;

a₉- all mixtures, in any proportion, composed of at least two of aforementioned components(a₁,a₂,a₃,a₄,a₅,a₆,a₇, a₈);

(b) a viscosity regulator in a concentration of 0-5%;

(c) a nonionic, anionic, cationic or ampholyte emulsifier in a concentration of 0-15%;

(d) a pH regulator, preferably a buffery system or an alkali in a concentration 0 -5 %;

(e) other dispersed systems of polymers and/or oligomers in a concentration of maxim 10%;

(f) other components in a maximum concentration of 2%;

(g) a solvent or a system of solvents, so that $a+b+c+d+e+g=100$.

2. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 1 which utilizes as an active component colloidal silica ;

3. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1 which utilizes as an active component alkali silicate (lithium silicate, sodium silicate, potassium silicate) or a mixture of them;

4. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1 which utilizes as an active component ammonium organic quaternary silicates with the general formula $((R_1R_2R_3R_4)N)_2O \times nSiO_2$ where R_1, R_2, R_3, R_4 may be identic or different radicals to 25 carbon atoms, resulting from alkane, alkene, cycloalkane, arene with or without lateral catena, alcohol, amine, halogen derivative, esters;

5. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1 which utilizes as active component monoalkoxysilanes, dialkoxysilanes, trialkoxysilanes or their mixtures;

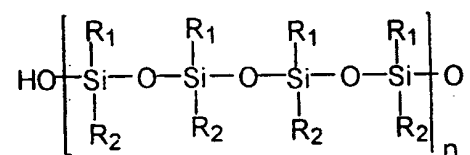
6. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 5, which utilizes as active component trialkoxysilanes or mixtures of trialkoxysilanes represented by the formula $RSi(OR')_3$ where R is a monovalent organic radical which has up to 25 carbon atoms and results from an alkane, cycloalkane, alkene, arene with or without lateral chain, alcohol, halogen derivative, alcohol, amine, ester and R' is a rest alkyl having to five carbon atoms.

7. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 5, which utilizes as active component dialkoxysilanes or mixtures of dialkoxysilanes represented by the formula $RR''Si(OR')_3$ where R' has the same significance as before, and R and R'' are identic or different monovalent organic radicals. R has the aforementioned significance (Claim 6), and R'' is also a monovalent radical, which meets the same conditions as R.

8. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 5, which utilizes as active component monoalkoxysilanes or mixtures of monoalkoxysilanes represented by the formula $RR'R'''SiOR'$, wherein R, R', R'' have

the aforementioned significance (Claims 6, 7), and R''' is also a monovalent radical, which meets the same conditions as R and R'' being identical with or different from them.

9. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which utilizes as active component siloxanes or mixtures of siloxanes represented by the formula:



wherein R_1 and R_2 represent identical or different radicals meeting the same conditions as R in claim 6.

10. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which utilizes as active component silicones or mixtures of silicones represented by the formula $\text{RSiO}_a(\text{OR})_b$, where R and R' represent identical or different organic radicals (coming from an alkane, cycloalkane, arene (with or without lateral chain) to maximum 25 carbon atoms which may also have substitutes (halogen, hydroxyl, amino, ester); a, b real numbers between 0 and 3, preferably a between 0,75 and 1,5, and b between 0,01 and 0,07, and the molecular mass of the silicone being more than 4000 u.a.m. and containing free groups of hydroxyl at the ends of silicone chain.

11. Colloidal emulsion shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which utilizes as active component alkyl orthosilicates corresponding to the formula: $\text{Si}(\text{OR})_4$, where R is a monovalent radical, identical or different, resulted from an aliphatic hydrocarbon (alkane, cycloalkane) to nine carbon atoms.

12. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which utilizes as active component salts of aforementioned silanols, siloxanes, siloxanols;

13. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 1, which uses as active component any combination, in any proportion, consisting of minimum two of the aforementioned components $a_1, a_2, a_3, a_4, a_5, a_6, a_7, a_8$;

14. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 1, which uses as component functioning as a viscosity regulator:

- ♦ cellulose derivatives: carboxymethylcellulose, methylcellulose, hydroxyethyl cellulose, etc;
- ♦ polyvinyl alcohol;
- ♦ polyacrylates and polymethacrylates alkali salts like: sodium polyacrylate, sodium polymethacrylate, etc;
- ♦ sodium and ammonium salts of carboxylate polymers;
- ♦ silanol and siloxanol salts like sodium methylsilicone;

15. Colloidal emulsion shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which uses as component (c) an emulsifier nonionic, anionic, cationic or an ampholyte:

16. Colloidal emulsion shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 15, which uses as component c an nonionic emulsifier, chosen from:

- ♦ alcohol ethers – where alcohol hydrocarbon rest may have linear or ramified catena and 7-25 C atoms and the number of ethylene oxid units is under 50;
- ♦ phenoethers
- ♦ A-B-A compounds, where each A represents an oligo rest or a polyester of a hydrocarboxylic acid, and B is a chain of polyoxoalkylene type;
- ♦ emulsifiers containing sulfoxi groups described in patent GB 2077620 ;
- ♦ polyoxyethylenesters of fatty acids;
- ♦ polyoxyethylenalkylethers and polyoxyetylenalkylphenylethers;
- ♦ partially esterified polyglycerol;
- ♦ natural substances like: lecithin, lanolin, saponin, cellulose alkylated derivatives with at most 4 C atoms in alkyl rest;
- ♦ polyoxyethylenglycols modified in conformity with GB-P No. 2,093,783, DE-PS No.3,109,317, U.S. Pat. No. 4,258,169, U.S. Pat. No. 4,257,995, DE-PS No. 2,932,175, DE-OS No. 3,111,562, either modified with esters of phosphoric acid (DE-PS No. 19,456) or modified with sulfonic acid (EP 19486);
- ♦ ethoxy products of monoalcohols, monoamines, monomercaptans, alkylmonophenols, in conformity with DE-OS No. 1,081,225;

- ♦ cyclic emulsifiers in conformity with DE-OS No. 3,062,328 / DE-OS No. 3,060,545;
- ♦ amines and amides derived from fatty acids;
- ♦ esters of fatty acids with alcohols, ethylenglycol, polyethylenglycol, glycerine, polyglycerine, propylenglycol, sorbitol, pentaerythritol, saccharose;
- ♦ polyethylenglycol condensation products (or polyethylenglycol oligomers) with alcohols, thioalcohols, fatty acids, amines and amides of fatty acids, polypropylenglycol, ethylenediamine, alkylphenols, condensed phenols, abietic acid, condensed amines and polysiloxane;
- ♦ propylenglycol condensation products with alcohols, fatty acids, and their derivatives (amines, amides, esters);
- ♦ higher alcohols with 8-20 C atoms;
- ♦ hydroxyethylcellulose, methylcellulose, gelatine, polyoxyethylenepolypropylen glycolether, salts of aminated lignin;
- ♦ polyvinylalcohol with a polymerization degree to 5000;
- ♦ glycolic ethers with hydrocarbon radicals having 8-20 C atoms, resulting from an alkyl rest or aryl one;
- ♦ colofony esters with glycerol;
- ♦ copolymers of ethylene oxide or propylene oxide;
- ♦ alkylpolyglycolethers having to 50 units resulted from ethylene oxide and alkyl rest to 20 C atoms;
- ♦ alkylarylpolyglycolethers with at most 50 units resulted from ethylene oxide and with an alkyl rest or aryl rest to 20 C atoms;
- ♦ ethylene oxide or propylene oxide oligomers with 200 units maximum;
- ♦ addition products of ethylene oxide or propylene oxide at amines with alkyl radical at most 20 C atoms;
- ♦ carboxylic acids with 6-24 C atoms per molecule;
- ♦ alkylpolyglycosides with formula R-O-Zi, where R is a saturated, aliphatic or aromatic, radical with 6-20 C atoms, and Zi is an oligosaccharide: with at most 10 units of monosaccharide;
- ♦ esters of sugars with α -hydroxycarboxylic acids and lactides in conformity with DE 19829305;
- ♦ polyoxyethylenglycols and monoalkylethers of polyoxyethylenglycol;

- ♦ cyclic emulsifiers in conformity with DE-OS 3062328, 3060545;
- ♦ polyoxypropylenglycol reaction products with ethylene oxide;
- ♦ polyoxyethylenpolyoxypropylenglycol soluble in water;
- ♦ natural substances or derivatives like: lecithin, lanolin, saponin, cellulose, carboxyalkylcellulose with alkyl rest of 5 C. atoms at most, etc;
- ♦ modified organopolysiloxanes containing at most 40 units ethylene oxide per molecule:
- ♦ organopolysiloxanes modified, in conformity with US 6060619, by the reaction of carboxyl group from the siloxane molecule with the hydroxyl group of the lactic acid.

17. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 15, which uses as component c an anionic emulsifier, chosen from:

- ♦ alkaline salts (Na, K) of alkylbenzene sulfonic acid;
- ♦ alkyl-sulfates with hidrocarbon rest of 5-20 C atoms and alkylethers-sulfates containing to 40 units of ethylene oxide or propylene oxide;
- ♦ alkaline salts (Na, K) of casein;
- ♦ algin acid and alginates of alkaline metals or alkaline earths;
- ♦ polyphosphates of alkaline metals or alkaline earths;
- ♦ sulfatate alcohols, type R-OSO₃Na, where R is an alkyl, aryl, or alkylaryl radical with 5 to 25 carbon atoms:
- ♦ sulfatate esters, where R is a radical having to 15 carbon atoms;
- ♦ esters or sulfosuccinic acid with monoalcohols or alkylphenols, where, optionally, monoalcohols or alkylphenols may be ethoxylated with 1-40 units of ethylen oxide or propylen oxide;
- ♦ alkaline or ammonium salts of alkyl, alkylaryl, arylalkyl phosphates with 6 to 20 C atoms in the hydrocarbon rest;
- ♦ alkyl, alkylaryl, arylalkyl phosphates (or their alkaline salts) with 6 to 20 C atoms in the hydrocarbon rest and containing to 40 units ethylene oxide or propylene oxid per molecule;
- ♦ ether alcohols and sulfonate ether phenols, where R is an alkyl, aryl, or alkylaryl radical with 5-25 C atoms;

- ♦ aliphatic sulfonic acids with a hydrocarbon chain, to 25 C atoms long, where R may still contain stable functional groups at hydrolysis in aqueous medium;
- ♦ sulfonic acids of dialkylated naphthalene;
- ♦ alkylaryl sulfonates with a hydrocarbon chain, where R is saturated or unsaturated hydrocarbon rest of maximum 25 C atoms:
- ♦ sulfonic esters of dicarboxylic acids;
- ♦ anionic emulsifiers, type alcoylate, described in W09907673 (EP.1005452);
- ♦ esters of higher acids with hydroxysulfonic acids;
- ♦ acyl derivatives (amides) of superior acids with aminosulfonic acids;
- ♦ phosphatides;
- ♦ alkaline earths or ammonium salts of carboxylic acids with 6-20 C atoms, where the hydrocarbon rest may be alkyl, aryl, or arylalkyl.

18. Colloidal emulsion shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 15, which uses as component c an cationic emulsifier or an ampholytic, chosen from:

- ♦ salts of primary, secondary, tertiary, amines containing 8-24 C atoms radicals with acetic acid, sulfuric acid, hydrochloric acid, phosphoric acid;
- ♦ quaternary alkyl- and alkylbenzeneammonium salts, which has alkyl group from 6 to 24 carbon atoms, with acetic acid, sulfuric acid, hydrochloric acid, phosphoric acid.
- ♦ alkylamines with 6-20 C atoms;
- ♦ ammonium quaternary salts of alkylarylamines or arylalkylamines with at most 24 C atoms per molecule;
- ♦ salts of alkylpyridine, alkylimidazole, alkylloxazole with alkyl rest of at most 20 C atoms, with acetic acid, sulphuric acid, hydrochloric acid, phosphoric acid.;
- ♦ salts of alkylpolyamines, alkylimidazoles;
- ♦ amino acids substituted, or not substituted, having at most 25 C atoms per molecule;
- ♦ betaine, alkylbetaine and alkylimidazole betaine with alkyl radical with at most 18 C atoms.

19. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 1,

which uses as component d a pH regulator, preferably a buffery system or a base in order to maintain the emulsion pH value between 7 and 11,5.

20. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 19, which uses a component functioning as a pH regulator chosen from: alkaline carbonates or bicarbonates (sodium or potassium), sodium phosphate, ammonium acetate, alkaline polyphosphates, sodium or potassium hydrophosphate, sodium molybdate, diethylamine, ethylenediamine, monoethanolamine, triethanolamine, morpholine, 2-amino-1-propanol, etc.

21. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which uses as (h) dispersed system of polymers or oligomers of these type: epoxy, phenol, polyester, vinylester, polyurethane, polyamide, furan, acrylic, polyethylene, polypropylene, polystyrene, polyvinylacetate resins.

22. Colloidal emulsion-shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claim 1, which uses as f) component, fungicide, bactericide, colouring, catalytic, aromatic corrosion inhibiting substances in a maximum 2% m.p.;

23. Colloidal emulsion shaped composition applied to eliminate ascensional humidity from building elements and to consolidate them, according to claims 1, which uses as g component d water or ethyl alcohol.

24. Procedure of application of a composition in order to fight off the dampness, characterized by the fact that, for stopping the water transport by capillarity, the building element is impregnated with the emulsion prepared as in claims 1, the emulsion being injected in the wall through orifices made either in one side or both side of the building element (as in fig. 1,2,3,4,), the distance among orifices being correlated with the emulsion diffusion radius around the orifice, so that no area among orifices remain unimpregnated with the emulsion.

Referințe bibliografice

Brevet RO nr. 93978

Brevet Ro 64969

Brevetul Ro 91966

Brevetul Ro 75304

Brevet Ro 107266

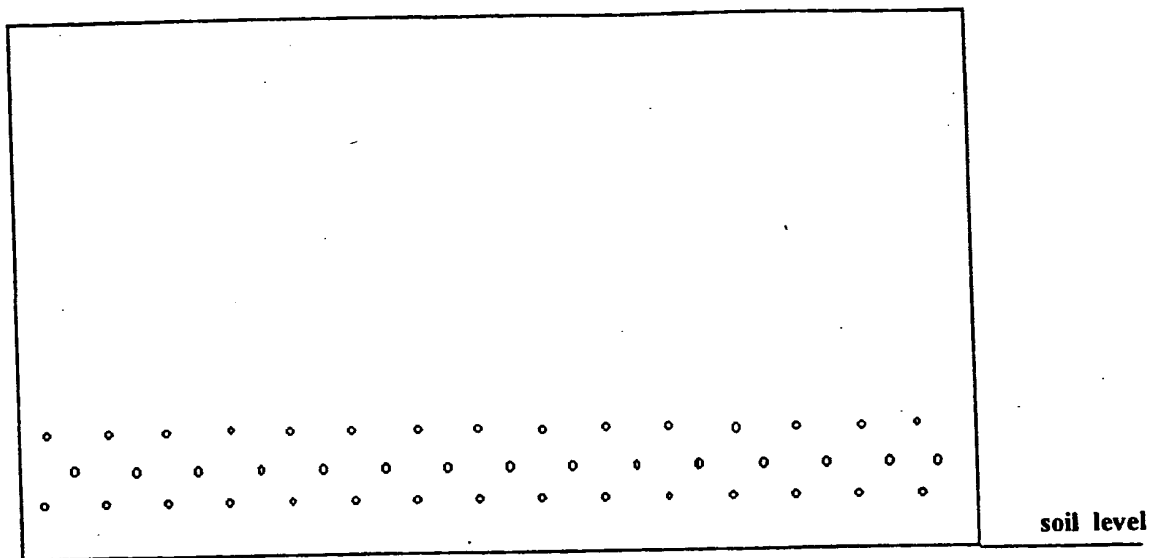


Fig. 1

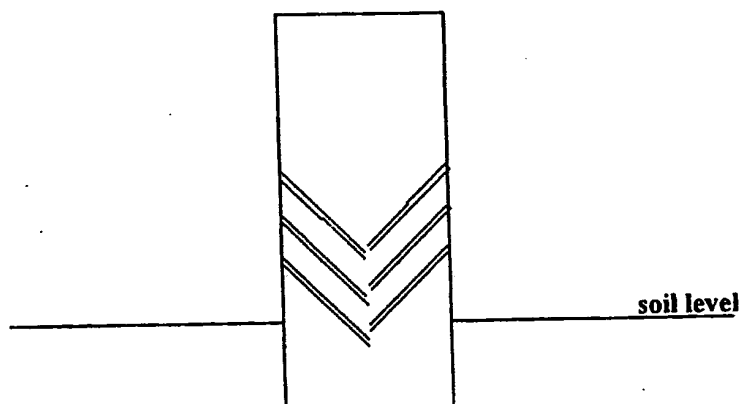


Fig 2

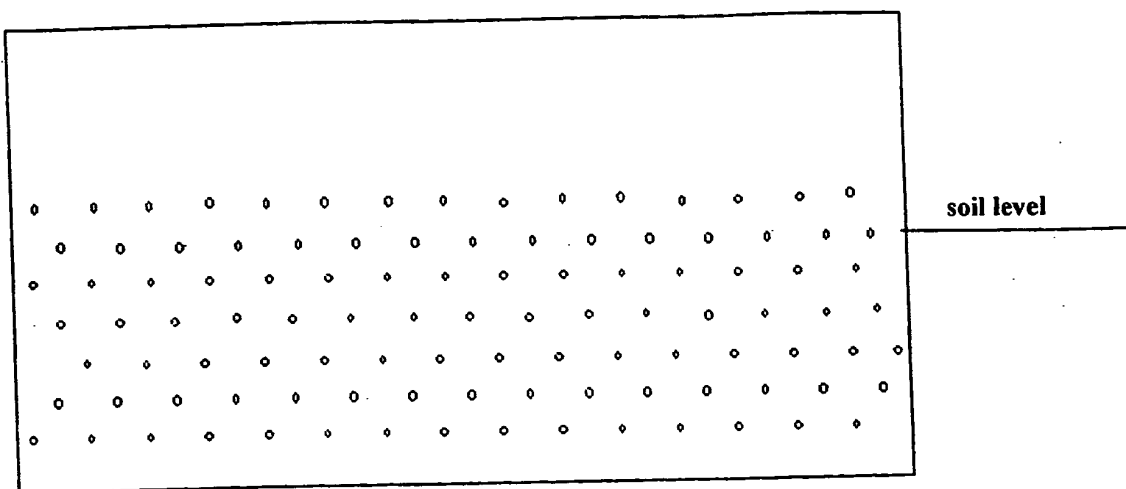


Fig 3

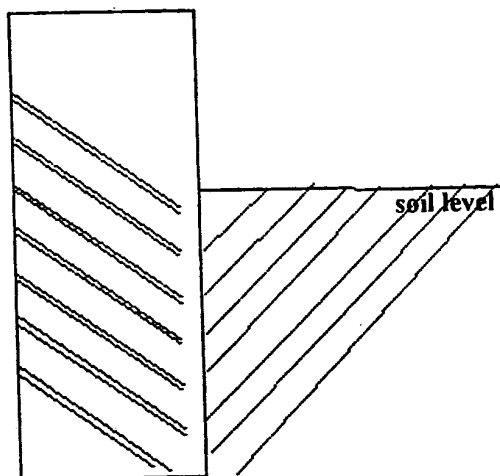


Fig 4